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The Structure at 198 K of (1*R*,5*R*,15*R*,16*R*)-5-Isopropenyl-2-methyl-1-[*N*-(*trans*-2-phenylcyclohexyloxycarbonyl)amino]-2-cyclohexene

BY VINCENT M. LYNCH, H. KENAN YASER, JAMES K. WHITESSELL AND BRIAN E. DAVIS

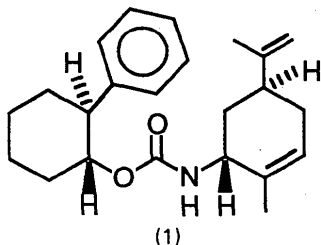
Department of Chemistry and Biochemistry, University of Texas at Austin, Austin, TX 78712, USA

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Abstract. *trans*-2-Phenylcyclohexyl *N*-(5-isopropenyl-2-methyl-2-cyclohexan-1-yl)carbamate, $C_{23}H_{31}NO_2$, $M_r = 353.50$, orthorhombic, $P2_12_12_1$, $a = 8.813$ (2), $b = 9.043$ (2), $c = 25.643$ (5) Å, $V = 2043.6$ (8) Å³, $Z = 4$, $D_x = 1.15$ g cm⁻³ (198 K), Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å, $\mu = 0.6734$ cm⁻¹, $F(000) = 768$, $T = 198$ K, $R = 0.0547$ for 1772 reflections [$F_o \geq 4\sigma(F_o)$]. Molecules are H-bonded into infinite columns parallel to **a**. The H bond involves the NH group and the carbonyl O atom of the carbamate moiety with relevant parameters: N11—H11...O13 (related by $\frac{1}{2} + x, \frac{1}{2} - y, -z$); N...O 2.910 (5), H...O 2.11 (5) Å, N—H...O 159 (4)°.

Experimental. The carbamate (1) was prepared from the adduct formed from (*R*)-limonene with the *N*-sulfinylcarbamate of (1*R*)-*trans*-2-phenylcyclohexanol (Whitesell & Carpenter, 1987; Whitesell, Carpenter, Yaser & Machajewski, 1990) by reaction with hexamethyldisilazane followed by thermal rearrangement (Yaser, 1990). X-ray structural analysis

was used to confirm the stereochemistry of the chiral center bearing nitrogen (C1) and independently confirmed by internal comparison with the absolute stereochemistry of the *trans*-2-phenylcyclohexanol used (Whitesell & Lawrence, 1986). Crystals were obtained by slow evaporation from hexanes. The data crystal was a clear, colorless needle of approximate dimensions 0.11 × 0.11 × 0.29 mm; the data were collected on a Nicolet R3 diffractometer using a graphite monochromator and a Nicolet LT-2 low-temperature delivery system; lattice parameters were obtained from the least-squares refinement of 40 reflections with $20.2 < 2\theta < 23.5^\circ$; ω scan technique with a 2θ range from 4.0 – 52.5° and a $1.2^\circ \omega$ scan at 5 – $10^\circ \text{ min}^{-1}$ ($h = -11 \rightarrow 11$, $k = -12 \rightarrow 12$, $l = 0 \rightarrow 32$). Two symmetry equivalent octants of data were collected (hkl and $-h, -k, l$) yielding a total of 4788 reflections of which 2417 were unique ($R_{\text{int}} = 0.0198$); four reflections (114, 132, 124, 125) were remeasured every 96 reflections to monitor instrument and crystal stability; a smoothed curve of the intensities of these check reflections was used to scale the data; the scaling factor ranged from 0.9912–1.014; the data were also corrected for Lp effects and absorption (based on crystal face measurements; transmission factor range was from 0.9832–0.9866). The data reduction, absorption and decay correction were applied using the Nicolet XRD *SHELXTL-Plus* software package (Sheldrick, 1988); reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (645



reflections); the structure was solved by direct methods (Sheldrick, 1988) and refined by full-matrix least squares using *SHELX76* (Sheldrick, 1976); 328 parameters were refined; non-H atoms were refined with anisotropic thermal parameters; all H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. However, the geometry of the H atoms on methylene carbons, C4 and C19, methyl carbon, C7, methine carbon, C16 and phenyl carbons, C24 and C25 did not refine well and were idealized (C—H, 0.96 Å). The U for H25 was fixed at $1.2 \times U_{eq}$ of C25. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = (0.5kI)^{-1/2}\{[\sigma(I)]^2 + 0.02I^2\}^{1/2}$; the intensity, I , is given by $(I_{peak} - I_{background}) \times (\text{scan rate})$; 0.02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects, absorption and decay; $\sigma(I) = [(I_{peak} + I_{background})^{1/2} \times (\text{scan rate})]$. The final $R = 0.0547$ for 1772 reflections, with $wR = 0.0607$ ($R_{all} = 0.0772$, $wR_{all} = 0.0651$) and a goodness of fit = 1.686; the maximum $|\Delta\rho| < 0.1$ in the final refinement cycle and the minimum and maximum peaks in the final ΔF map were -0.18 and 0.26 e Å^{-3} , respectively. Differentiation between enantiomorphs could not be made on the basis of the X-ray diffraction results ($wR = 0.0607$). The scattering factors for non-H atoms were taken from Cromer & Mann (1968), with anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970); the scattering factors for H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55).^{*} Figures were generated using *SHELXTL-Plus* (Sheldrick, 1988). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labeling scheme is shown in Fig. 1. Other computer programs used in this work are listed in reference 11 of Gadol & Davis (1982).

Related literature. Derivatives of 2-phenylcyclohexanol have been utilized as chiral auxiliaries in exerting stereochemical control of reaction products (Whitesell, Chen & Lawrence, 1985; Whitesell & Carpenter, 1987; Whitesell, Carpenter, Yaser & Machajewski, 1990).

^{*} Tables of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles involving the H atoms, torsion angles, structure factor amplitudes and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53894 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters (\AA^2) for the non-H atoms of (1)*

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C1	0.2984 (6)	0.1895 (6)	−0.0735 (2)	0.036 (2)
C2	0.3407 (5)	0.3262 (5)	−0.1039 (2)	0.0341 (14)
C3	0.3902 (5)	0.3160 (5)	−0.1528 (2)	0.0376 (15)
C4	0.4207 (6)	0.1734 (5)	−0.1797 (2)	0.043 (2)
C5	0.4447 (5)	0.0463 (5)	−0.14197 (15)	0.0341 (14)
C6	0.3098 (6)	0.0458 (6)	−0.1040 (2)	0.039 (2)
C7	0.3207 (6)	0.4708 (6)	−0.0769 (2)	0.045 (2)
C8	0.4731 (6)	−0.0966 (6)	−0.1705 (2)	0.042 (2)
C9	0.6288 (8)	−0.1081 (8)	−0.1946 (2)	0.056 (2)
C10	0.3709 (9)	−0.2017 (7)	−0.1751 (2)	0.057 (2)
N11	0.3831 (4)	0.1821 (5)	−0.02413 (13)	0.0373 (12)
C12	0.3128 (5)	0.171 (5)	0.0213 (2)	0.0320 (13)
O13	0.1773 (3)	0.1725 (4)	0.02821 (12)	0.0466 (11)
O14	0.4165 (3)	0.1684 (3)	0.06123 (10)	0.0361 (9)
C15	0.3603 (5)	0.1461 (5)	0.11346 (15)	0.0336 (14)
C16	0.4127 (5)	−0.0049 (5)	0.13287 (15)	0.0365 (15)
C17	0.3706 (6)	−0.0238 (6)	0.1902 (2)	0.040 (2)
C18	0.4351 (7)	0.0995 (6)	0.2244 (2)	0.047 (2)
C19	0.3804 (6)	0.2486 (5)	0.2042 (2)	0.046 (2)
C20	0.4229 (6)	0.2706 (6)	0.1478 (2)	0.041 (2)
C21	0.3559 (5)	−0.1328 (5)	0.1002 (2)	0.0370 (15)
C22	0.2022 (6)	−0.1464 (6)	0.0874 (2)	0.044 (2)
C23	0.1498 (7)	−0.2696 (7)	0.0616 (2)	0.056 (2)
C24	0.2469 (8)	−0.3798 (7)	0.0479 (2)	0.068 (2)
C25	0.4003 (8)	−0.3699 (7)	0.0596 (2)	0.067 (2)
C26	0.4537 (7)	−0.2453 (6)	0.0850 (2)	0.050 (2)

Table 2. *Bond lengths (\AA) and angles ($^\circ$) for the non-H atoms of (1)*

1	2	3	1–2	1–2–3
C2	C1	C6	1.508 (7)	114.7 (4)
C6	C1	N11	1.520 (7)	111.7 (4)
N11	C1	C2	1.472 (6)	110.9 (4)
C3	C2	C7	1.329 (6)	122.5 (4)
C3	C2	C1		120.8 (4)
C7	C2	C1	1.491 (7)	116.7 (4)
C4	C3	C2	1.487 (7)	123.8 (4)
C5	C4	C3	1.517 (6)	112.7 (4)
C6	C5	C8	1.536 (7)	115.7 (4)
C6	C5	C4		107.4 (4)
C8	C5	C4	1.505 (7)	111.3 (3)
C1	C6	C5		112.0 (4)
C9	C8	C10	1.509 (8)	122.4 (5)
C9	C8	C5		114.2 (5)
C10	C8	C5	1.315 (9)	123.4 (5)
C12	N11	C1	1.322 (5)	121.6 (4)
O13	C12	O14	1.207 (5)	123.3 (4)
O13	C12	N11		126.4 (4)
O14	C12	N11	1.373 (5)	110.3 (3)
C15	O14	C12	1.442 (5)	118.0 (3)
C16	C15	C20	1.525 (6)	111.2 (3)
C16	C15	O14		108.9 (3)
C20	C15	O14	1.532 (7)	107.9 (4)
C17	C16	C21	1.526 (6)	111.6 (4)
C17	C16	C15		109.9 (4)
C21	C16	C15	1.514 (6)	113.8 (3)
C18	C17	C16	1.528 (7)	112.4 (4)
C19	C18	C17	1.523 (7)	109.4 (4)
C20	C19	C18	1.507 (7)	111.4 (4)
C15	C20	C19		111.4 (4)
C22	C21	C26	1.399 (7)	118.2 (5)
C22	C21	C16		121.1 (4)
C26	C21	C16	1.388 (7)	120.6 (4)
C23	C22	C21	1.375 (8)	120.5 (5)
C24	C23	C22	1.361 (9)	120.5 (6)
C25	C24	C23	1.388 (10)	120.6 (6)
C26	C25	C24	1.385 (8)	119.0 (6)
C21	C26	C25		121.1 (6)

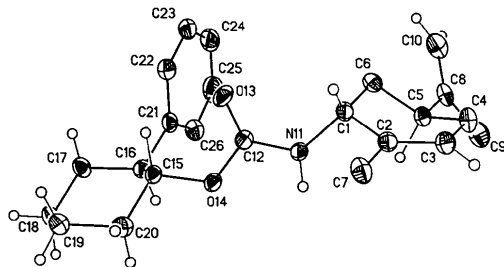


Fig. 1. View of (1) showing the atom-labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. Methylene H atoms at C4 and C6, methyl H atoms at C7 and C9 and all phenyl ring H atoms have been omitted for clarity. Other H atoms are represented as spheres of arbitrary size.

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Structure of 6 α -Chloro-3,20-dioxo-4-pregnen-17 α -yl Acetate

BY ZDZISLAW WAWRZAK AND PAWEŁ GROCHULSKI

Technical University of Łódź, Institute of Physics, ul. Wolczanska 219, 93-005, Łódź, Poland

ZDZISLAW GALDECKI

Technical University of Łódź, Institute of General Chemistry, ul. Zwirki 36, 90-924 Łódź, Poland

AND JANE F. GRIFFIN, PHYLLIS D. STRONG AND WILLIAM L. DUAX

Medical Foundation of Buffalo, Inc., 73 High Street, Buffalo, NY 14203, USA

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Abstract. C₂₃H₃₁ClO₄, $M_r = 406.95$, orthorhombic, $P2_12_12_1$, $a = 13.155(2)$, $b = 13.397(2)$, $c = 12.369(2)$ Å, $V = 2179.9(9)$ Å³, $Z = 4$, $D_x = 1.24$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 2.05$ cm⁻¹, $F(000) = 872$, $T = 293$ K, $R = 0.064$, $wR = 0.063$ for 2828 observed reflections with $F_o > 3\sigma(F_o)$. Rings *B* and *C* have chair conformations and the *D* ring is in an intermediate sofa-half-chair conformation. Ring *A* assumes an intermediate sofa-half-chair conformation and is flat relative to the rest of the steroid skeleton. The progesterone side chain has a conformation typical for other 17 α -ester steroids; the C(16)–C(17)–C(20)–O(20) torsion angle is $-23.0(5)^\circ$.

Experimental. Crystal with dimensions $0.2 \times 1.2 \times 1.4$ mm, Nicolet *P3* diffractometer, cell dimensions

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and Laue symmetry from 25 centered reflections ($27 < 2\theta < 31^\circ$) checked with oscillation photographs, Mo $K\alpha$ radiation, Nb filtered, no monochromator, scan width $2.4^\circ + 1.04(2\theta_{K\alpha 2} - 2\theta_{K\alpha 1})$, scan speed from 3 to $30^\circ \text{ min}^{-1}$ in 2θ , $2\theta_{\text{max}} = 60^\circ$, $0 \leq h \leq 19$, $0 \leq k \leq 19$, $0 \leq l \leq 18$, 4254 reflections were measured using θ – 2θ scan mode, 3588 of them were unique, $R_{\text{int}} = 0.026$. Four standard reflections (10,0,0; 080; 006; 565) were measured every 196 reflections and varied in intensity by $\leq 5\%$ during the data collection. Intensity corrections made with the *DREAM* program (Blessing, 1987).

Direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed positions of all non-hydrogen atoms. The positional and anisotropic displacement parameters of all non-hydrogen atoms were refined by the full-